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BLOW-OUT OF RUBBER BLOCKS DUE TO INTERNAL HEATING

by

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Rubber blocks undergo explosive blow-out when heated in a microwave oven. The process appears to be the same as the blow-out caused by repeatedly-applied, severe compressive loads. It is attributed to high internal pressures generated by a volatile product of thermal decompostion.

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Bursting took place at lower temperatures for softer compounds and for compounds that softened markedly upon heating. The blow-out temperature was also quite different for different elastomers, being low for butyl rubber, about 180 degrees C. and somewhat higher for SBR and NBR compounds, over 200 degrees C. Polybutadiene compounds did not blow-out at all, probably because the products of thermal decomposition were less volatile.

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Blow-Out of Rubber Blocks Due to Internal Heating

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1. Introduction

When rubber is deformed, part of the work of deformation is transformed into heat. Because rubber is a rather poor conductor of heat, the internal temperature under repeatedly-applied deformations can reach high values, especially for thick articles. Tire treads and tank track pads can become so hot in the interior that they explode. This phenomenon is known as "blow-out." But the mechanism of failure is not well understood It has been attributed to mechanical fatigue [1], and biaxial tensile fracture aggravated by high temperature [2,3]. In contrast, it is attributed here to high internal pressure of volatile substances, sufficient to burst open the surrounding material. The volatiles are thought to be a product of thermal decomposition. We show that blow-out can be produced by direct heating of rubber compounds in a microwave oven, and to some extent by internal heating of rubber sheets by embedded resistance heating wires, without any mechanical stressing.

2. Experimental details

Vulcanized rubber cylinders, 25mm high and 17.5mm in diameter, and sheets of various thicknesses were made in a compression mold using conventional recipes. A typical mix formulation was as follows, in parts by weight: SBR 1502, 100; carbon black (N330, Vulcan 3, Cabot Corp.), 50; zinc oxide, 5; stearic acid, 2; processing oil (Sundex 790T, Sun Petroleum Co.), 5; sulfur, 1; accelerator (Santocure MOR, Monsanto Co.), 0.5. Vulcanization was effected by heating for 50 min at 150°C.

Values of Young's modulus E were determined from stress-strain relations in compression, at ambient and elevated temperatures.

Cylindrical specimens were subjected to repeated compression using a Goodrich Flexometer, as described in ASTM D623 - 67 [4]. The frequency of loading was 30 Hz and the double-amplitude of oscillation was 6.35mm. Static compressive loads of either 32 or 43 kg were applied to the sample by attaching weights to the machine beam. Experiments were terminated when the sample failed by blow-out, typically within 15 min. Measurements of blow-out temperature were made by means of a probe thermocouple inserted into the sample immediately after blow-out.

Other samples were heated in a microwave oven without imposing any deformation. Blow-out similar to that obtained with the Goodrich Flexometer occurred in less than one minute.

Some vulcanized sheets, about 6mm thick, were heated by passing an electric current through a fine straight copper wire, about 125mm long and 160 μm in diameter, embedded in the center of the sheet before vulcanization. The power input was adjusted to heat the sample at about the same rate as in the microwave oven, 3-5°C/sec. Internal temperatures were determined from the measured resistance of the wire and the temperature coefficient of resistance of copper. At temperatures between 200 and 300°C, the rubber sheet showed surface discoloration, followed soon afterwards by appearance of a blister, which is considered equivalent to blow-out.

Thermogravimetric analysis was carried out on some compounds and the volatiles released by blow-out were isolated and analyzed by gas chromatography and mass spectrometry.

3. Results and discussion

(i) Blow-out from repeated compression and solely from heating

Blow-out temperatures for Goodrich flexometer samples are given in Table 1. They reached values of 200°C or more, suggesting that blow-out is a consequence of thermal decomposition of the polymer.

Blow-out temperatures for samples subjected to direct heating in the microwave oven were approximately the same, Table 1, and the appearance of the failed samples was also quite similar, Figure 1. Thus, the criterion for blow-out seems to be a simple one: the interior of the sample must reach a critical temperature. Mechanical working is only a means of generating heat internally; it is not essential for blow-out.

Significant differences were noted in the appearance of samples. Flexed samples became barrel-shaped, due to compression set, Figure 1, and decomposition was concentrated in the center. In contrast, microwave samples decomposed over larger internal regions, wherever severe heating had occurred.

(ii) Blow-out temperatures for typical rubber compounds

Blow-out temperatures are given in Table 1. They ranged from about 160°C to about 250°C, depending upon the polymer and the mix formulation.

Two general features were noted. First, the blow-out temperature was higher for harder materials. For example, compounds SBR2 and SBR3 had different amounts of sulfur and accelerator, while SBR1 and SBR3 differed in carbon black content, giving vulcanizates of the same polymer with markedly different elastic modulus. Blow-out took place at higher temperatures for the higher modulus compounds.

Values of Young's modulus E at temperatures close to the

blow-out temperature are included in Table 1; they confirm this general conclusion. Moreover, this feature is consistent with the proposed mechanism of blow-out. The critical pressure at which a small internal cavity in a rubber block will expand indefinitely is given approximately by $5E/6$ [5,6]. Thus, a harder material would be expected to withstand higher internal pressures without rupturing.

Some materials appeared to soften markedly at high temperatures and undergo blow-out at the same time. For example, butyl rubber compounds turned into a soft, buttery consistency at the blow-out temperature, about 180°C. In contrast, NBR and SBR compounds were found to be stiffer than others initially and to retain their stiffness at high temperatures. They were found to have higher blow-out temperatures also.

Natural rubber compounds became soft and sticky as a result of thermal decomposition, whereas NBR and SBR compounds remained relatively dry. In both cases, however, blow-out took place explosively, especially for NR materials with C-C or monosulfide crosslinks (NR2 and NR4) which blew out at relatively high temperatures, Table 1.

Polybutadiene compounds did not undergo blow-out, even after long periods of heating in the microwave oven. Instead, the samples softened, swelled, and became porous in the interior, Figure 2. Apparently, the vapor pressure of the products of decomposition is significantly lower for this polymer. "Blow-out" temperatures were taken as the temperatures at which pronounced swelling occurred.

Thus, two factors appear to govern the susceptibility of rubber compounds to blow-out. The first is their propensity to generate high-vapor-pressure volatiles. The second is their

tendency to soften on heating and thus lose resistance to expan-
sion of a pressurized internal cavity.

(iii) Possible nature of volatiles causing blow-out

Certain ingredients in rubber formulations would be expected
to volatilize at high temperatures and create a high internal
pressure. For example, if water is present, high internal pres-
sures would be generated at temperatures much above 100°C. In
order to test this possibility, 0.5 percent of salt was included
in several mix formulations and the vulcanized samples were
soaked in water for several days before testing, so that they
absorbed 1-2% by weight of water. Blow-out took place at
virtually the same temperature as for dry samples, indicating
that absorbed water is not the main cause of blow-out.

Another possible volatile ingredient of many rubber com-
pounds is a hydrocarbon processing oil. However, when an other-
wise identical formulation was prepared with no processing oil,
blow-out took place at about the same temperature as before.
Thus, the processing oil itself was not responsible for blow-out.

Another possibility is dissolved air. Samples that had been
stored in vacuo for four weeks were found to have significantly
higher blow-out temperatures, by about 30°C, Table 2. On exposing
them to air again for several weeks, the blow-out temperatures
were reduced almost back to the original values. These results
suggest that air is at least partially responsible for blow-out,
probably by catalyzing thermal decomposition.

Different formulations are known to differ in susceptibility
to thermal decomposition. For example, vulcanizates with poly-
sulfide crosslinks will soften at lower temperatures than those
with monosulfide or with carbon-carbon crosslinks because of the
lower strength of such linkages. If crosslink rupture is also

accompanied by the production of a volatile substance, then blow-
out will take place when a significant degree of crosslink
rupture occurs. In accord with this hypothesis, peroxide
recipes, yielding C-C crosslinks, and monosulfide ("sulfurless")
recipes gave the highest blow-out temperatures, Table 1,
approaching the temperatures at which polymer decomposition would
be expected. And differences observed between elastomers can be
attributed to differences in stability of the crosslinks formed
within them.

Thus, although the exact nature of the volatile constituent
is not clear, the hypothesis that it is a gaseous product of the
decomposition of crosslinks accounts for the main features of
blow-out, namely, the general temperature range in which it
occurs, its close relationship with softening temperatures, the
lower blow-out temperatures for materials containing polysulfidic
crosslinks in comparison with C-C crosslinks, and the marked
differences observed between different polymers.

(iv) Decomposition by heating with an embedded resistance wire

Temperatures of surface discoloration are given in Table 1.
Blistering occurred somewhat later, at temperatures of the order
of 500°C. These temperatures are much higher than those for
blow-out but they are considered to be over-estimates of the
actual rubber temperature because of heat losses through the
rubber and severe decomposition of rubber in the immediate
vicinity of the wire.

Decomposition temperatures measured in this way were found
to be less reproducible than microwave blow-out temperatures and
they depended strongly on the rate of heating. Thus, although
the wire-heating method is appealingly simple, it proved to be
less satisfactory than microwave heating for determining

susceptibility to thermal decomposition.

(v) Unresolved issues

Measured blow-out temperatures were surprisingly low, at most 250°C, whereas rapid thermal decomposition was not found by TGA and by direct heating with resistance wires below about 300°C. It is possible that the blow-out temperatures were too low, because of delay in inserting the thermocouple and difficulty in obtaining good thermal contact with fractured rubber surfaces. Improved experimental techniques are needed to determine blow-out temperatures precisely.

A second unknown is the nature of the volatiles. Vapors emitted by blow-out samples were collected and analyzed. They were found to consist of low-molecular-weight hydrocarbons, including monomer, but no substance characteristic of the particular crosslinking system could be detected. And yet large differences were found in blow-out temperatures for different vulcanizates of the same polymer. Again, improved experimental techniques are needed to determine the decomposition products completely.

4. Conclusions

Explosive "blow-out" of thick rubber blocks under repeatedly-applied, severe compressive loads is due entirely to the development of high internal temperatures. If the compound is electrically conductive, the phenomenon can be duplicated in a microwave oven without imposing any mechanical loads.

The principal cause of blow-out is the generation of a volatile decomposition product. Pressurized cavities within the rubber then expand and burst. Bursting is easier in some compounds because they soften markedly at high temperatures and thus lose resistance to cavity expansion.

Different elastomers have strikingly different blow-out

temperatures. Butyl rubber compounds blow out at relatively low temperatures, about 180°C, whereas NBR and SBR compounds blow out at temperatures of about 200°C or higher. Polybutadiene compounds did not blow out at all. Instead, they developed internal cavities that grew in size and number but never burst open to the exterior, probably because the vapor pressure never reached sufficiently high levels.

Acknowledgements

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Table 1: Flexometer and Microwave Blow-out Temperatures T_b, T_b,
and Discoloration and Blistering Temperatures T_d, T_{bl} (°C)
for Electrically Heated Rubber Sheets

Material	E(MPa) at 25°C	E(MPa) at 100°C	T_b Flexometer	T_b Microwave	T_d Discoloration	T_{bl} Blistering
NR1	4.5	2.2	180	175±15	235±15	350±50
NR2 (monosulfide cure)	6.1	4.5	--	215	--	--
NR4 (peroxide cure)	4.5	4.3	--	222±7	--	--
NR5	--	--	--	--	340±15	525±35
SBR1	4.5	3.4	--	196±8	--	--
SBR2	3.6	--	--	173±10	--	--
SBR3	9.1	4.5	215	228±10	217±25	--
SBR4 (peroxide cure)	4.5	5.1	--	252±5	--	--
BR1	1.3	--	--	200±3	295±75	410±40
IIR1	7.7	1.2	162±2	182±4	--	--
IIR2	6.3	0.8	164±2	185±5	385±50	450±50
NBR1	9.6	5.5	--	256±10	--	--
NBR2	4.2	3.1	--	242±15	--	--

Table 2: Effect of Removing Air on Blow-out Temperatures (°C)

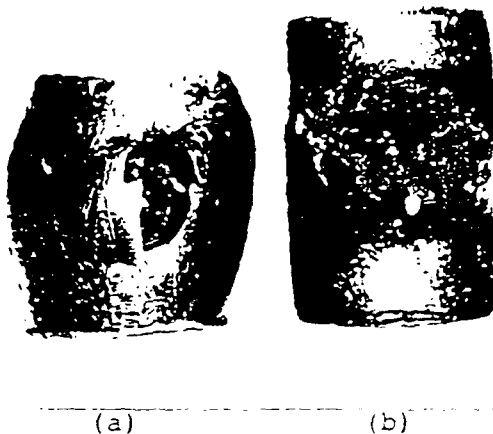| Material	As Molded	After Degassing	After Standing in Air
NR1	195±8	220±4	184±4
NR4 (peroxide cure)	225±8	257±4	240±5
SBR3	240±10	280±10	250±7
IIR2	193±7	195±7	140±10

(a)   (b)

Figure 1.   Samples of NR2 after blow-out.   (a) Flexometer,

(b) Microwave heating.

Figure 2.   Cross-section or PB sample after microwave heating.

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